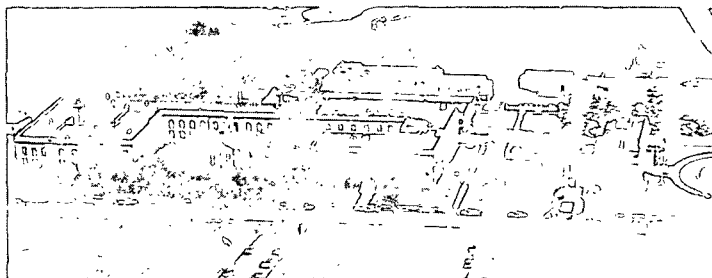


NOV 2 1976



**THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN**

**IPC TECHNICAL PAPER SERIES  
NUMBER 38**

**CORRELATION OF FIBER MORPHOLOGICAL VARIATION AND WET  
MAT COMPRESSIBILITY OF LOBLOLLY PINE BLEACHED KRAFT PULP**

**A. P. BINOTTO AND G. A. NICHOLLS**

**SEPTEMBER, 1976**

Correlation of fiber morphological variation and wet mat compressibility of loblolly pine bleached kraft pulp

A. P. Binotto and G. A. Nicholls

Foreword

Due to great interest in the genetic improvement of trees, the many studies undertaken in this area include TAPPI committee activities concerned with the aim of assigning economic values to specific methods of altering wood and fiber properties. From such studies and activities, it is apparent that significantly more is known about the relationships of fiber morphology and products than is known about fiber morphology and processes. The subject of this paper is a part of the area of fiber morphology and processes.

This study shows that fiber morphological variation, which relates to growth within a tree, can result in changes of about 30% in fiber wet mat compressibility at 5-15% consistency, as encountered in displacement bleaching and washing. The variation is such that, if more juvenile wood is utilized, it probably would result in lowering the capacity <sup>and</sup> of increasing the cost of the equipment. When utilizing more mature wood the converse would apply.

This paper has been submitted for publication in Tappi.

Correlation of fiber morphological variation and wet mat compressibility of loblolly pine bleached kraft pulp

A. P. Binotto and G. A. Nicholls

Abstract

Fiber fractions were characterized by differences in mean fiber length,  $\underline{L_f}$ , and wall fraction, calculated from measurements of mean fiber width,  $\underline{d_f}$ , and wall thickness,  $\underline{WT}$ . Assuming a simplified model, moments of inertia for flattened fiber cross sections,  $\underline{I_F}$ , were calculated using  $\underline{d_f}$  and  $\underline{WT}$  data and increased with increase in fiber length for earlywood and latewood. Fiber fractions gave wet mat density,  $\underline{c} = \frac{MP_f N}{P_f}$  for static pressure,  $\underline{P_f} = 10-90$  cm H<sub>2</sub>O. A single value was found for the compressibility constant,  $\underline{N}$ , and the compressibility constant,  $\underline{M}$ , correlated with  $\underline{L_f}/\underline{d_f}$  and  $\underline{L_f}$ , for all fiber fractions. For any two fiber fractions,  $\underline{dc}/\underline{dP_f}$  would provide a more reliable indication of relative compressibility than determination of the compressibility constant " $\underline{M}$ ."  $\underline{M}$  was found to be proportional to  $(1/\underline{I_F})^{1/3}$ , thereby implying that flattening of cross sections of these fibers is part of the compressibility mechanism with  $\underline{WT}$  as a predominant morphological factor. Results imply that inclusion of more juvenile wood would increase wet mat compressibility, whereas more mature wood would decrease it. With stated assumptions, this could result, for example, in respectively decreasing or increasing the capacity of displacement bleaching and washing equipment.

---

A. P. Binotto, Graduate student, and G. A. Nicholls, Professor Pulp and Paper Technology, The Institute of Paper Chemistry, Appleton, Wisconsin 54911.

## INTRODUCTION

Due to great interest in the genetic improvement of wood and fiber properties during recent decades, various groups around the world have undertaken many studies in this area. One comprehensive study on the relationship between fiber morphology and kraft paper properties for loblolly pine was used as part of the basis for TAPPI committee activities concerned with the aim of assigning economic values to specific methods of altering wood and fiber properties (1). From such studies and activities it is apparent that there is significantly more known about the relationships of fiber morphology and products than is known about fiber morphology and processes.

There have been some studies in which the structures of model fibers have been related to properties of interest in pulping and papermaking processes. For example, the effects of glass or synthetic fiber dimensions and shape on compression and the resistance to flow of fluids through fiber mats have been reported (2-5). On the basis of such studies it is to be expected that correlations might exist between wet mat compressibility, as reviewed by Han (6), and certain aspects of wood fiber morphology. One aspect is that thick-walled latewood fibers with relatively high wall fraction and thin-walled earlywood fibers with relatively low wall fraction would vary significantly in the compressibility of their wet mats. The latewood fibers, which are known to have a significantly greater elastic modulus (7-9), might be expected to give lower wet mat compressibility. Another possible correlative aspect would be changes in fiber length to diameter ratio. In

addition it is necessary to take into account degree of delignification since this also relates to mat compressibility and flow of fluids through fiber mats.(10,11).

The aim of this study is to obtain correlations of wet mat compressibility and wood fiber morphological variation, and to increase current understanding of the mechanism of compressibility.

## RESULTS AND DISCUSSION

### Morphological Fractions

Bleached loblolly pine kraft pulp was prepared, separated into earlywood and latewood fractions using revolving tilted bowls like those described by Jacquelin (12), and then further classified into a range of fiber lengths as in the Experimental part. An indication of the separation into earlywood and latewood is given by the data in Table I on wall fraction (the percentage of the fiber radius that consists of fiber wall), derived from measured mean fiber widths and wall thicknesses. The wall fraction of 30-32% for earlywood, compared with 62-68% for latewood fiber fractions, provided two distinct sets of fibers.

[Table I here]

Earlywood and latewood are further subdivided in Table I according to mean fiber length. The fiber length range of 1.6-3.9 mm for earlywood is about comparable to that of 1.7-4.1 mm for latewood and in both sets 50 to over 80% of the fibers were whole. Also, the major variations in fiber characteristics are in wall fraction for between-sets and in fiber length for within-sets. For within-sets, increases in fiber width and wall thickness accompany an increase in fiber length, but these trends

generally will be referred to in terms of fiber length which is the most accurately determined dimension. These joint increases in fiber dimensions are typical of unbroken wood pulp fibers (13,14) and are unlike the model fibers studied previously.

### Moments of Inertia

Consideration of approximate models for compressibility by other workers (6) leads to the following equation:

$$\underline{c} = \left[ \frac{3\pi^{1/3} \rho_f^5 \underline{d}_f^4}{4^7 \alpha (1+\beta) \underline{c}_o^2 \underline{IE}^{1+\beta}} \right]^{1/3} \underline{P}_f^{(1+\beta)/3} = \underline{MP}_f \underline{N} \quad (1)$$

where:  $\underline{c}$  = mat density

$\underline{c}_o$  = initial mat density

$\underline{d}_f$  = fiber diameter

$\underline{E}$  = modulus of elasticity

$\underline{I}$  = moment of inertia

$\underline{M}, \underline{N}$  = compressibility constants

$\underline{P}_f$  = static pressure

$\alpha, \beta$  = constants associated with a load distribution function

$\rho_f$  = fiber density

In this equation,  $\underline{N}$  varies much less than  $\underline{M}$ , and  $\beta$  for unconditioned wood pulp mats is about  $\pm 0.1$  (6). Hence,  $\underline{IE}^{1+\beta}$  is mainly an expression of flexural rigidity, which in turn appears to be a significant component of  $\underline{M}$ .

By simply assuming the fiber wall is homogenous and of uniform mass distribution, the second moments of the areas or moments of inertia for

flattened cross sections,  $\underline{I_F}$ , can be calculated on the basis of a rectangular model, from the expression:

$$\underline{I_F} = \frac{2}{3} \underline{d_F} (\underline{WT})^3. \quad (2)$$

Using the measured values of  $\underline{d_F}$  and  $\underline{WT}$ , calculated values of  $\underline{I_F}$  listed in Table I show an increase with increase in fiber length for both earlywood and latewood, due to the above noted interrelation of fiber dimensions.<sup>1</sup>

Consideration of elastic modulus,  $\underline{E}$ , is confounded by the anisotropic nature of wood fibers and by increase in apparent elastic modulus with decrease in fibril angle, which has a high correlation with increase in fiber length (8,14,15). Further bewilderment in quantitatively comparing literature data arises, for example, from one basis relating to the relative elastic modulus of undelignified wood (16) and another basis relating to the wet and dry transverse compressibility of various pulp fibers (17). Nevertheless, in general, observed trends (7-9,14-17) qualitatively indicate that, if data on apparent elastic moduli were to be available for wet fiber fractions as in Table I,  $\underline{E}$  would tend to be greater for latewood than earlywood and would tend to increase with fiber length as does  $\underline{I_F}$ . Furthermore, there is no apparent reason to believe trends in  $\underline{E}$  might cause trends in flexural rigidity to depart significantly from those seen for  $\underline{I_F}$  in Table I.

<sup>1</sup>The case of moments of inertia for circular cross sections,  $\underline{I_C} = (\underline{r_o}^4 - \underline{r_i}^4)/4$ , where  $\underline{r_o} = \underline{d_F}/2$  and  $\underline{r_i} = (\underline{d_F}/2) - \underline{WT}$ , was also considered and found to be inapplicable, as discussed later.

### Compressibility

Replicate compressibility data for each of the morphological fractions were used in log-log plots of wet mat density,  $\underline{c}$ , vs. static pressure,  $\underline{P}_f$ , to obtain individual best-fit curves by linear regression. The slopes of these curves are the primed values for the compressibility constant,  $\underline{N}$ , in Table II. This also lists the corresponding primed values for the intercept or compressibility constant,  $\underline{M}$ . Although slopes were determined by linear regression, the values are sensitive to the exact positioning of the best-fit curve and normal variability in the experimental data results in uncertainty over what value should be used in practice. After further analysis, a weighted arithmetic mean value of  $\underline{N}$  was determined for all of the data, then curves with that slope were best-fitted to the data for each fiber fraction. This gave the unprimed values for  $\underline{N}$  and  $\underline{M}$  as in Table II.

[Table II here]

Confirmation of the applicability of these latter values to the actual experimental data is provided by linear scale plots in Fig. 1. Each curve is based on the calculation of  $\underline{MP}_f^{\underline{N}}$  using relevant  $\underline{N}$  and  $\underline{M}$  values so that:

$$\underline{c} = \underline{MP}_f^{\underline{N}} \quad (3)$$

and  $\underline{c}$  on any curve is the corresponding interpolated wet mat density.

[Fig. 1 here]

Curve smoothing within the 95% confidence limits of the experimental data, on the basis of the single value of 0.373 for  $\underline{N}$  and relevant  $\underline{M}$  values, is illustrated in Fig. 2 for the earlywood fractions. A similar



situation applies to latewood. The smoothed data are taken as more generally applicable to the experimental data on the whole. However, it is of interest to compare the set of calculated values for the slopes of curves,  $dc/dP_f$ , in Fig. 1 with the set in parentheses shown in Table II. The latter set is based on  $\underline{N}'$  and  $\underline{M}'$  in Table II. Both sets follow the trend seen for  $\underline{M}$  when  $\underline{N}$  is 0.373, whereas neither set follows the trend seen for  $\underline{M}'$ . For any two fiber fractions in this group, it would not necessarily be established that  $\underline{N}$  can be treated as constant in value, in which case  $dc/dP_f$  apparently would provide a more reliable indication of relative compressibility than the coefficient, " $\underline{M}$ ."

[Fig. 2 here]

Plots in Fig. 3 permit comparison of correlations of  $\underline{M}$ , with fiber length to diameter ratio,  $\underline{L}_f/\underline{d}_f$ , and fiber length,  $\underline{L}_f$ . In compressibility studies on glass and nylon fibers (6),  $\underline{L}_f/\underline{d}_f$  correlated well at a given pressure with mat density. This is proportional to  $\underline{M}$  in Eq. 3 when  $\underline{P}_f$  and  $\underline{N}$  are constant. The comparable correlation of  $\underline{M}$  vs.  $\underline{L}_f/\underline{d}_f$  (Fig. 3) and  $\underline{c}$  vs.  $\underline{L}_f/\underline{d}_f$  for glass and nylon fibers, studied previously, relates to the mechanism of compressibility, as discussed by Han. An interesting point in Fig. 3 is the similarity of the correlations of  $\underline{M}$  with either  $\underline{L}_f/\underline{d}_f$  or  $\underline{L}_f$ . This is believed to result from the length of wood fibers being naturally interrelated with other dimensions, including  $\underline{d}_f$ , as discussed above.

[Fig. 3 here]

The separation of the curves for  $\underline{M}$  vs.  $\underline{L}_f$  in Fig. 3 could be due to differences in  $\underline{d}_f$ ,  $\underline{I}$  and/or  $\underline{E}$  since, on the basis of Eq. 1:

$$\underline{M} \propto (\underline{d}_f^4/\underline{I}\underline{E})^{1/3} \quad (4)$$

Attempts to correlate  $\underline{M}$  and either  $(\underline{d}_f^4/\underline{I}_F)^{1/3}$  or  $\underline{d}_f$  failed to give meaningful results, as also found for the latter by Jones (2). However, it was discovered that  $\underline{M}$  is proportional to  $(1/\underline{I}_F)^{1/3}$  as in Fig. 4, and in view of the simplicity of the basis used to calculate  $\underline{I}_F$ , this result is outstanding. No such correlation was found between  $\underline{M}$  and calculated moments of inertia for circular cross sections,  $\underline{I}_C$ . Thus, an identifiable part of the compressibility mechanism for these fibers consists of a flattening of their cross sections, for which case Eq. 2 and the order of the data points in Fig. 4 show that wall thickness,  $\underline{WT}$ , is the pre-dominant morphological factor.

[Fig. 4 here]

As indicated above, elastic moduli,  $\underline{E}$ , would probably tend to have the same trends as  $\underline{I}_F$  data in Table I. Therefore,  $\underline{E}$  could be making at least an equally important contribution in the compressibility mechanism.

### Implications

From the above it is apparent that morphological variation which relates to growth within a tree (13) can result in changes of about 30% in wet mat compressibility at about 5-12% consistency (Table II), as encountered in displacement bleaching and washing. The trend of this relationship implies, for example, that if more juvenile wood which contains relatively more short fibers were included in a chip supply, wet mat compressibility would increase, whereas if more mature wood were included wet mat compressibility would decrease.

Thus, if more juvenile wood is utilized, it probably would be necessary in displacement bleaching and washing to use a lower maximum pressure between the washer head and screen to avoid any significant increase in

thickening. Assuming a constant dilution factor and that any changes in diffusion, dispersion, etc., are relatively insignificant, this need would result in lowering the capacity or increasing the capital cost of the equipment. When utilizing more mature wood the converse would apply.

## EXPERIMENTAL

### Pulp Preparation, Fractionation, and Morphological Characterization

A ca. 3-inch thick board was cut from the center of a 27-yr-old medium dense loblolly pine (dbh = 9 in.) and a board similarly was cut from each large slab on either side of the first board. The tree was from a natural even-aged experimental forest in Effingham County, Georgia. Chips from the boards, excluding pith, were kraft pulped using 20.6% active alkali at 170°C for 4 hours to give screened pulp kappa no. 19.8 (TAPPI Standard Method T 236 m-60). Bleaching was by CEDED with 6.0, 2.5, 1.0, 1.8, and 0.6% of relevant chemicals at 25, 60, 70, 60, and 70°C, for 60, 60, 165, 60, and 240 min, respectively. Chlorination consistency was 3.0%, others were 10% and final TAPPI brightness 89%.

The apparatus and procedure for obtaining flocs in the separation of bleached pulp into latewood and earlywood fibers were as described before (11) except consistency was 1%. Separation of a gently agitated suspension of flocs from field fibers was accomplished by syphoning suspended field fibers onto a muslin-covered box. Following the initial separation into floc and field fibers, each component was processed a second time to give floc-floc and field-field fibers. Scanning electron microscopy confirmed that these had typical latewood and earlywood fiber characteristics as illustrated previously (11). Earlywood and latewood fractions

were further separated by Bauer-McNett classification (approximately 600 runs; TAPPI Standard Method T 233 su-64) into on-65, on-20, and on-10 mesh fractions with increasing mean fiber length as in Table I. An unidentified equipment change, detected by percent retained data, resulted in two groups of on-65 mesh fibers for latewood.

Fiber width,  $\underline{d_f}$ , and cell wall thickness,  $\underline{WT}$ , were measured on 100 fibers at magnification 210X with a light microscope equipped with a filar micrometer. Fibers were wet mounted in water/glycerin with a cover glass placed gently on top so fibers, particularly earlywood, were not flattened. In calculating wall fraction, fiber width was used as diameter. Fiber lengths,  $\underline{L_f}$ , were determined on samples of 1,000 fibers using a semiautomatic recorder according to Illvessalo-Pfaffli and Alfthan (18). Percentage of whole fibers was determined by counting 200 fibers at magnification 35X.

#### Compressibility

Apparent wet mat density in g o.d. fiber per cc wet mat was determined as a function of compacting pressure using an established procedure (19). First compression data were taken at 15-min intervals.

#### LITERATURE CITED

1. van Buitjtenen, J. P., Alexander, S. D., Einspahr, D. W., Ferrie, A. E., Hart, T., Kellog, R. M., Porterfield, R. L., and Zobel, B. J., Tappi 58(9): 129 (1975).
2. Jones, R. L., Tappi 46(1): 20 (1963).

3. Elias, T. C., Tappi 50(3): 125 (1967).
4. Bliesner, W. C., Tappi 47(7): 392 (1964).
5. Labrecque, R. P., Tappi 51(1): 8 (1968).
6. Han, S. T., Pulp Paper Mag. Can. 70(9): 65 (1969).
7. Hardacker, K. W. The Physics and Chemistry of Wood Pulp Fibers. TAPPI STAP No. 8: 201 (1970).
8. Alexander, S. D., Marton, R., and McGovern, S. D., Tappi 51(6): 277 (1968).
9. Leopold, B. and Thorpe, J. L., Tappi 51(7): 304 (1968).
10. Gren, U. B., Svensk Papperstid. 75(19): 785 (1972).
11. Andrews, B. D. and Nicholls, G. A., Appita 27(6): 411 (1974).
12. Jacquelin, G., Tech. Rech. Paper. 7: 22 (1966).
13. McMillin, C. W., Wood Sci. Technol. 2: 166 (1968).
14. Panshin, A. J., DeZeeuw, C., and Brown, H. P., Textbook of Wood Technology. Chap. 3, p. 70, New York, San Francisco, Toronto and London, McGraw-Hill Book Company, 1964.
15. Mark, R. E., Cell Wall Mechanics of Tracheids. Chap. 7, p. 168-170, New Haven and London, Yale Univ. Press, 1967.
16. Yiannos, P. N. and Taylor, D. L., Tappi 50(1): 40 (1967).
17. Hartler, N. and Nyren, J. TAPPI Paper Physics Seminar, "The Physics and Chemistry of Wood Pulp Fiber," p. 76. Appleton, WI, May 1969; Tappi 53(5): 820 (1970).
18. Illvessalo-Pfaffli, M. S. and Alfthan, C. v., Paperi Puu 39: 11 (1957).
19. Ingmanson, W. L. and Andrews, B. D., Tappi 42(1): 29 (1959).

---

A portion of a thesis submitted by A.P.B. in partial fulfillment of the requirements of The Institute of Paper Chemistry for the degree of Doctor of Philosophy from Lawrence University, Appleton, WI 54911, Dec., 1976. The authors are grateful to G. A. Baum, N. L. Chang, S. T. Han, and R. W. Nelson for suggestions, to V. J. Van Drunen for his help and to other staff for various contributions.

Table I. Mean Fiber Dimensions<sup>a</sup> and Related Data

Wall fraction, % <sup>b</sup>	$\frac{d_f}{x 10^2 \text{ mm}^c}$	$\frac{WT}{x 10^2 \text{ mm}^c}$	$\frac{L_f}{\text{mm}^c}$	Whole fibers, %	$\frac{I_f}{x 10^8 \text{ mm}^4}$
<u>Earlywood</u>					
30 <sup>d</sup>	3.99 ± 0.25	0.59 ± 0.05	1.63 ± 0.02	51	0.6
32 <sup>e</sup>	4.50 ± 0.22	0.73 ± 0.07	3.05 ± 0.03	80	1.2
32 <sup>f</sup>	4.96 ± 0.23	0.80 ± 0.07	3.94 ± 0.04	89	1.7
<u>Latewood</u>					
62 <sup>d</sup>	3.24 ± 0.19	1.00 ± 0.05	1.74 ± 0.03	50	2.2
66 <sup>d</sup>	3.41 ± 0.17	1.13 ± 0.07	2.07 ± 0.04	59	3.3
66 <sup>e</sup>	3.57 ± 0.20	1.18 ± 0.06	2.98 ± 0.03	73	3.9
68 <sup>f</sup>	3.70 ± 0.17	1.26 ± 0.05	4.13 ± 0.06	85	4.9

<sup>a</sup>Arithmetic means.

<sup>b</sup>Calculated from fiber width,  $\frac{d_f}{\text{mm}}$ , and cell wall thickness,  $\frac{WT}{\text{mm}}$ , for various fiber lengths,  $\frac{L_f}{\text{mm}}$ .

<sup>c</sup>Mean ± 95% confidence limits.

<sup>d,e,f</sup>On-65, 20, and 10 mesh screens, respectively.

Table II. Compressibility Data for Mats of Different Fiber Fractions

Mean fiber length, mm	$\underline{N}'$	$\underline{M}' \times 10^3$ c.g.s. units <sup>a</sup>	$\underline{N}$	$\underline{M} \times 10^3$ c.g.s. units	$\underline{d} / \underline{dP}_f \times 10^7$ , g/cm-dyne <sup>b</sup>	
					$\underline{P}_f = 10 \text{ cm H}_2\text{O}$	
					$\underline{P}_f = 90 \text{ cm H}_2\text{O}$	
<u>Earlywood</u>						
1.6	0.361	2.02	0.373	1.79	21.0 (21.0)	5.3 (5.0)
3.1	0.390	1.43	0.373	1.70	19.9 (20.5)	5.0 (5.4)
3.9	0.376	1.61	0.373	1.66	19.5 (19.6)	4.9 (4.9)
<u>Latewood</u>						
1.7	0.373	1.65	0.373	1.64	19.2 (19.3)	4.8 (4.9)
2.1	0.376	1.55	0.373	1.60	18.8 (18.9)	4.7 (4.8)
3.0	0.363	1.73	0.373	1.56	18.3 (18.1)	4.6 (4.4)
4.1	0.366	1.69	0.373	1.57	18.4 (18.3)	4.6 (4.5)

<sup>a</sup>(g/cm<sup>3</sup>)/(dynes/cm<sup>2</sup>) $\frac{N}{N}$ .<sup>b</sup>Calculated using  $\frac{dc}{dP_f} = NMP_f^{(N-1)}$  or  $[N'M'P_f^{(N'-1)}] \dots (4)$ .

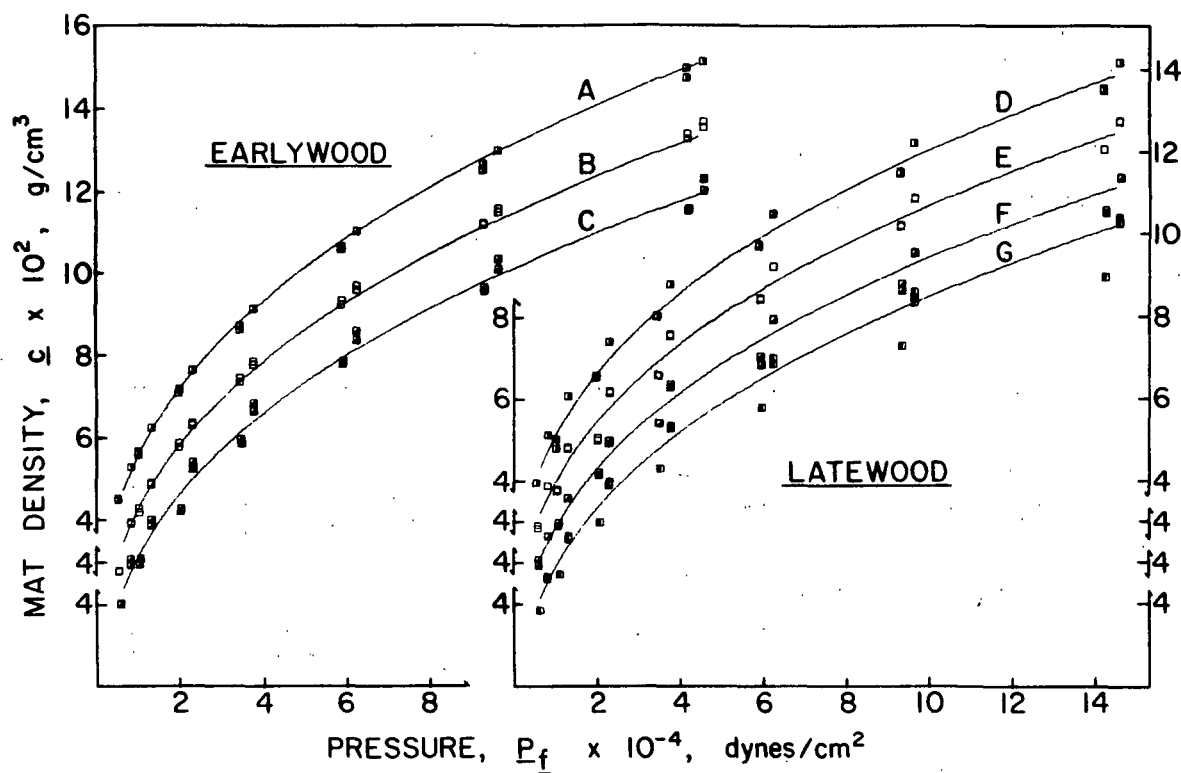


Fig. 1. Experimental data points for wet mat density at various static pressures for earlywood and latewood fiber fractions showing curves with wet mat density calculated from  $\frac{MP_f^N}{L}$ , with  $N = 0.373$  and  $M$  as in Table II. Curves A, B, C, D, E, F, and G are for mean fiber lengths of 1.6, 3.1, 3.9, 1.7, 2.1, 3.0, and 4.1 mm, respectively.



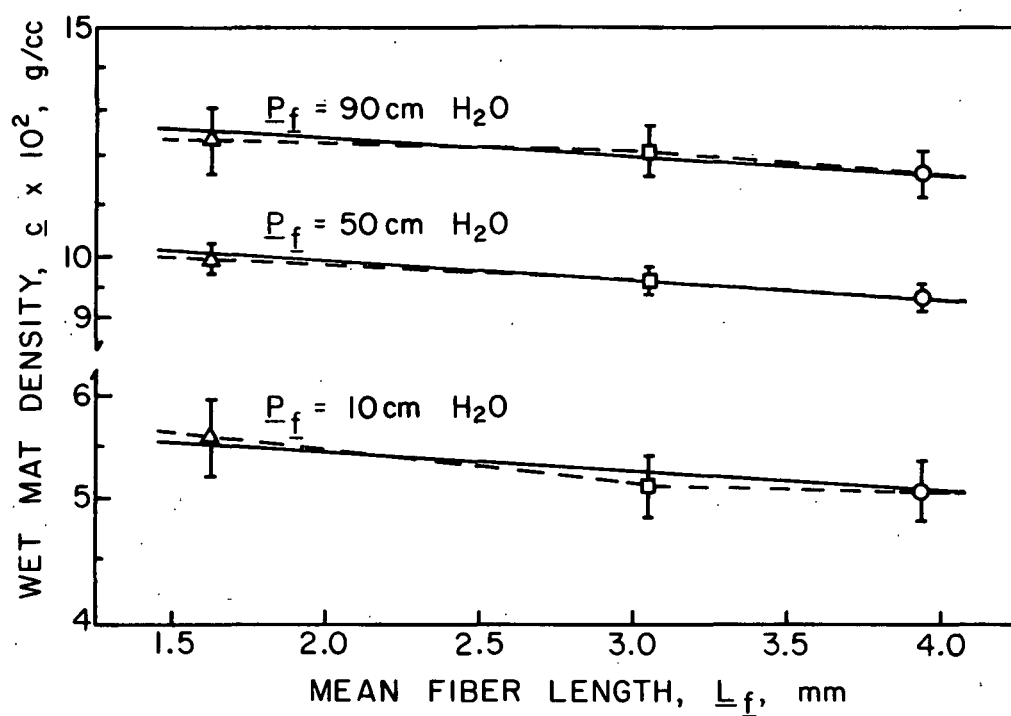


Fig. 2. Plots at three pressures showing mat density means with 95% confidence limits (corresponding compressibility constants are primed values in Table II) vs. mean fiber length for earlywood fractions. Solid line curves are based on unprimed compressibility constants  $\underline{N}$  and  $\underline{M}$  in Table II.

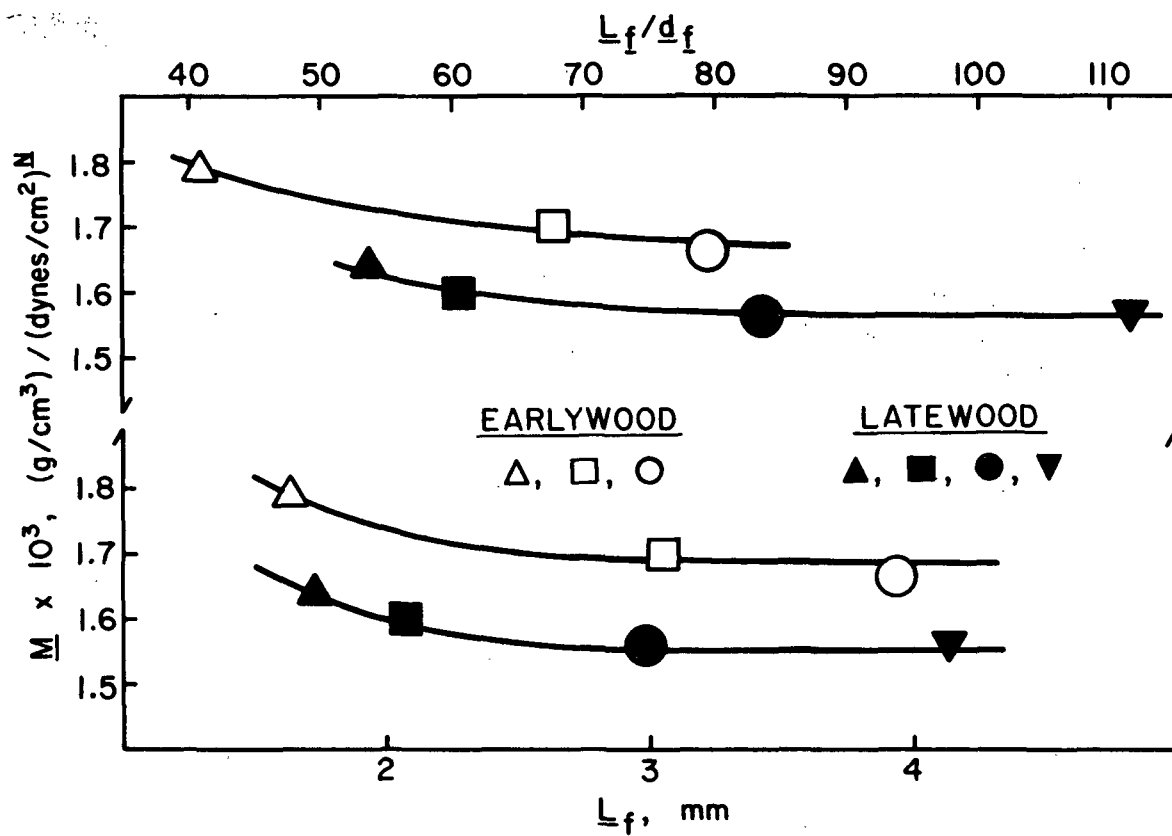


Fig. 3. Compressibility constant,  $\bar{M}$ , vs. fiber length to fiber diameter ratio,  $L_f/d_f$  (upper curves), and fiber length,  $L_f$  (lower curves).

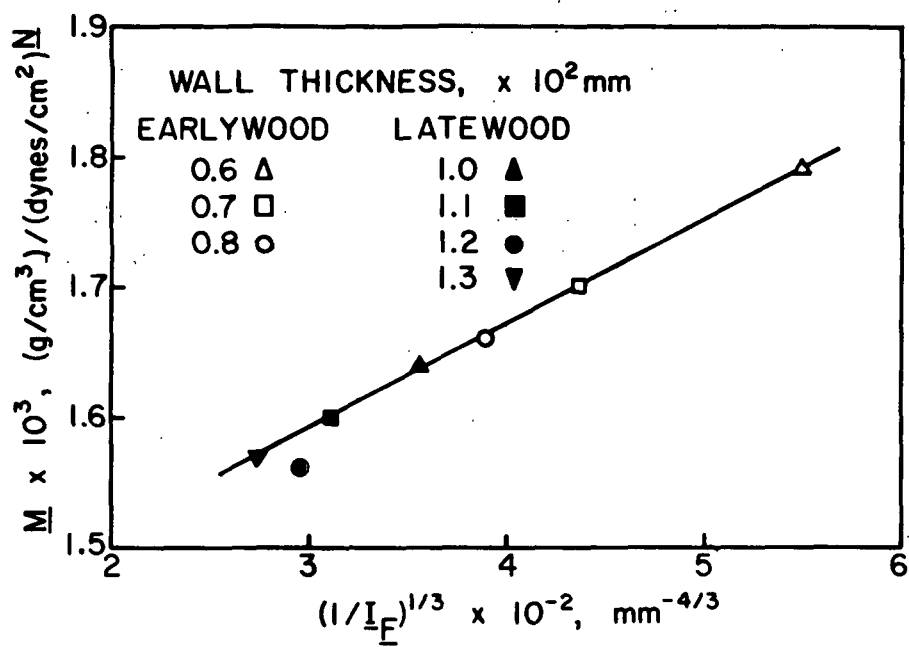


Fig. 4. Compressibility constant,  $\underline{M}$ , vs.  $(1/\underline{I}_F)^{1/3}$  for earlywood and latewood fiber fractions.